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DISPERSION STRENGTHENING

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One is justified, perhaps, in stating that important strides have been made in the past five to eight years in bringing oxide dispersion strengthened alloys nearer to commercial status; in fact, however, such commercial status is still not enjoyed in a meaningful sense. To a smaller extent there has been a better understanding of processing variables in alloy production, but then only in the very simple alloy systems, notably in those alloys not complexly alloyed and not containing chromium in amounts capable of achieving oxidation resistance.

To an important degree, one can state that our understanding of the mechanisms of strengthening is still inadequate, and very little effort has gone into studies to develop such understanding. The scramble appears to have been in alloy development, perhaps rightly so, but not to such an over-riding extent. Deformation and fracture studies have seen too little support. Evaluation of the material in terms of fatigue behavior, impact strength over a wide temperature range including cryogenic temperatures, and effects of low temperatures on ductility require much more extensive study.

The purpose of this chapter is to examine some of the progress which has been made in a number of areas, and to assess the growth and potential of oxide and other hard particle dispersion strengthened alloys in this light.

Mechanisms of Strengthening

1) Oxide Particle Size and Shape - The literature appears to support, without argument, the desirability of obtaining hard particles as fine as 50 to 100 Å⁰ (1,2). The finest particles measured and reported for experimental alloys have been of that size⁽²⁾. The particles were produced in dilute copper-aluminum alloys by internal oxidation. The particle size of thorium in TD-nickel has also been reported to be of that order of magnitude.

Mechanical blending, selective oxide reduction in an oxide mixture, and salt decompositions techniques have generally been unable to produce oxide particles finer than about 200 - 500 Å (0.02 - 0.05 micron) on average, even though it is definite that some of the particles are finer than 0.02 micron.

Even internal oxidation will not yield particles much finer than 0.02 - 0.05 micron unless conditions are ideal and many variables are controlled: in particular:

a) the free energy of formation of the solvent (matrix) metal must be very low relative to that of the oxidizable solute element; thus the combination Cu-Be works very much better than Ni-Al for production of ultra-fine oxides;

b) the solute content must be low, usually in an amount to yield less than about 3 volume percent of oxide;

c) the diffusion distance for oxygen should be short; the smaller the better, even down to near micron dimensions;

d) the solubility for oxygen in the solvent metal must be relatively high.

Very little is known regarding hard particle shape, and very little has been published on the subject^(3,4). Where shapes of oxide particles are significantly different than equi-axed polygonal or spherical, comparisons are not available for the same alloys and compositions.

Only in the case of zirconia was there an effort to note the relative benefits of polygonal versus flake particles. The polygonal zirconia - copper alloy had superior properties to the one containing flake zirconia⁽³⁾

As of now, it is impossible to predict, for example, what benefits, if any, are to be derived through the use of oxide fibers or filaments (or other hard particle shapes). There are frequent inquiries regarding the merits of whiskers, fibers, and filaments of various length to diameter

ratios as the dispersed phases in metallic (crystalline) matrices. While an answer cannot be given without necessary qualifications, it would appear that for a small volume content of the dispersoid (under about 10 percent) there is little likelihood that improvements can be realized through the use of shapes other than near-polygonal types, given a particular minimum particle dimension. There will be further discussion of this below.

2) Particle Spacing - The important role of particle spacing has probably enjoyed the maximum amount of unanimity; yet the agreements are more in kind than in amount and direction. Basically, two measures of particle spacing are being widely used, namely, interparticle spacing and mean free path⁽⁵⁾. Depending on the alloy in question and its structural characteristics, it has been demonstrated frequently that both measures often produce useful relationships with properties^(2,5,6,7). It is unfortunately true that other structural variables, such as volume content of oxide in a single alloy series, also relates well to properties⁽⁶⁾; as a result it is difficult to evaluate the role of structure sharply.

Of great interest is the role of volume fraction of oxide on strength values both at low and elevated temperatures. For dispersion strengthened alloys several investigators have shown that volume fraction of the hard phase is an important variable^(2,8). Because these studies were performed on totally different alloys, one with bonded⁽⁸⁾ and the other with unbonded hard particles⁽²⁾, comparisons are difficult and re-inforcement of arguments is meaningless. It is unfortunate that the volume content of the hard phase was restricted to low values in these studies, because it is expected that volume fraction might have its greatest meaning at higher volume contents, for example, above 5 to 10 percent.

As an experimental technique, it is extremely difficult to measure the particle spacing meaningfully. Particles finer than about 0.05 micron,

particularly when unbonded by the matrix, pull out in polishing and leave enlarged holes. Values of particles spacing are instead obtained by indirect means; knowing the particle size and volume fraction of oxide phase, one assumes a uniform distribution (which seldom exists) and calculates an average particle spacing value. Clearly direct measurements are needed before further significant progress can be made in this area.

3) Stored Energy of Deformation - The biggest disagreements, the greatest speculation, and the most poorly defined or measured quantities are associated with the amount and role of "equivalent cold work" retained by the structure as a result of the work it undergoes in processing. In early developments in the field the usual procedure was to depend on an oxide content varying from 5 to 20 volume percent and to strive for extrusion ratios of 5 to 30 in terms of reduction of area^(1,3,4,7). When oxide particle size and distribution were suitable, very high values of creep and rupture resistance were achieved; recrystallization temperatures were very high (within a few degrees of the melting temperature for aluminum⁽⁹⁾ and copper⁽²⁾, and within about 100°C in the case of nickel^(10,11,12); room temperature strength properties were only fair, at best.

It has variously been suggested that there is bonding between the oxide and the matrix; that there are possibly coherency strains around oxide particles when they are finer than 100 Å; and that the strength is dependent on the shear resistance of the fine hard particles⁽²⁰⁾. Direct tests of these hypotheses are not easy to perform or to interpret, and related indirect data are presented counter to these theses.

It has been the contention of the author, and his co-workers, that the observed strength, both at low and high temperatures, is essentially the strength gained as a result of the cold work experienced by the alloy during processing^(5,9,13,14,15).

Table I, for example, shows the effect on room temperature strength of dilute additions of aluminum to copper; internal oxidation of the same alloy, but without cold work; internal oxidation followed by cold work; and internal oxidation of fine powders followed by hot extrusion, typical of the processing of oxide dispersion strengthened alloys^(2, 21). These results are compared to pure annealed copper, and 50 percent cold worked copper. It is obvious that the dilute additions of aluminum in solid solution, and internal oxidation without cold work, do not result in properties equal to the strength properties of cold worked copper. Instead, 50 percent cold work in copper gives about the same strength values as cold worked, internally oxidized copper, however the cold work is introduced. It should be clear that if coherency strains were present, the internally oxidized Cu-Al alloys would be vastly stronger than they are.

TABLE I
Yield Strength at 80° F of Various Copper Alloys

Composition	Condition	Y. S. (0.2%) psi after internal oxidation	Y.S. (0.2%)psi internal oxidation plus cold work
Pure Cu	annealed **	7,800	-
Pure Cu	50% cold reduction **	-	64,000
Cu-0.22% Al+	wire	30,000	70,000
Cu-0.44% Al+	wire	35,000	70,000
Cu-3.5% Al ₂ O ₃	powder extrusion*	-	65,000

* Cu-0.77% Al alloy in powder form, internally oxidized, then extruded at 1380°F (750°C) (2)

+ reference 21

** not internally oxidized

The interesting work of Tracey and Worn⁽¹⁶⁾ offered, apparently, the first documented experimental evidence that one could just as readily introduce the cold work after hot working the metal-metal oxide compact. Their extrusion ratios were low, resulting in little equivalent cold work. The use of low oxide content, with good distribution, followed by judicious selection of cold work and intermediate annealing steps, yielded a product with improved formability and fabricability.

Whereas it had been possible to achieve 66 percent cold reduction with an aluminum - 7 volume percent Al_2O_3 alloy and 29 percent with an aluminum - 14 volume percent Al_2O_3 alloy on continuous rolling⁽⁹⁾, Tracey and Work report a capability to achieve greater than 90 percent cold reduction with a 3 volume percent ThO_2 alloy of nickel. Bufferd⁽¹⁷⁾ has recently indicated that it was possible to introduce 8 steps of 10 - 15 percent cold reduction per step with intermediate annealing treatments below the recrystallization temperature of the alloy; his alloy was an iron - 3 volume percent ThO_2 alloy, extruded initially at a low extrusion ratio. With each step, as was the case with Tracey and Work, there was an increase in both low and high temperature strength, accompanied by a small increase in ductility.

Rasmussen and Grant⁽¹⁵⁾ have recently proposed a possible mechanism for the strengthening process during cold work, with and without intermediate annealing treatments. Figure 1 shows the change in "particle spacing" as a result of thermo-mechanical working. The apparent inter-particle spacing decreases with increasing cold work; the role of the intermediate annealing step is to sweep the dislocation into low angle boundaries, permitting further cold work with less danger of cracking, and with improved ductility.

The role of the oxide then remains that of inhibiting grain boundary shear and migration plus the pinning of dislocation tangles. In no way does the oxide contribute directly to the strength of the alloy.

Several additional items of research lend strong support to this conclusion.

Edelson and Baldwin⁽¹⁸⁾ showed, for a series of composite, sintered (to about 98 percent density) binary composites that copper was not strengthened by second phase particles unless there was bonding or wetting of one phase for the other. Accordingly, Al_2O_3 and chromium, both insoluble in copper, did not increase the room temperature yield strength for additions up to 20 percent, whereas iron, which is bonded to copper, did result in an increase in yield strength. Even though the particle sizes of the excess phases were relatively coarse, the results were unambiguous in their story.

Murphy⁽¹⁹⁾ in unpublished work, has shown that when Ni(Ti) solid solution alloy powders are internally carburized (akin to the internal oxidation of Ni (Ti) solid solution alloys⁽¹¹⁾), for conditions wherein the volume fraction of TiC phase in Ni was approximately equivalent to the amount of TiO_2 in Ni, and the particle size of the TiO_2 was finer than the TiC, the room temperature strength properties of the Ni-TiC alloy were almost twice those of the Ni- TiO_2 alloy (140,000 psi yield strength versus about 70,000). The important difference appears to be the contribution of the bonding energy between Ni and TiC compared to the absence of bonding strength in the Ni- TiO_2 system.

While it is not possible to evaluate in a quantitative way the contribution of bonding energy to strength, either for coherent or non-coherent systems, it is clear that the absence of bonding in the oxide case results in no contribution to strength at lower temperatures compared to bonded composite alloys. At high temperatures the resistance to recovery

and recrystallization of the stable oxide dispersion strengthened alloys results in a relative strengthening compared to conventional alloys where solution and growth of particles leads to a rapid loss of strength which is clearly observed in longer time tests.

A completely unknown entity in considerations of strength in oxide or hard particle dispersion strengthened alloys is the role of texture. In early work with SAP alloys, it was observed that the 7 percent Al_2O_3 alloy had a more strongly preferred structure than the 14 percent Al_2O_3 alloy⁽⁶⁾; for both alloys the extrusion ratio was 20 to 1 or larger. In recent work on Ni-Mo- ThO_2 alloys⁽¹⁵⁾ containing 7 to 9 volume percent of ThO_2 , with a high extrusion ratio once more, there was a noted absence of a preferred orientation of the structure. In contrast, heavy cold work of hot pressed SAP alloys, in Russian work⁽²²⁾, resulted in a decided texture which was presumed to lead to an increase in strength in the working direction.

Using the low oxide content alloys (about 3%), Tracey and Worn's⁽¹⁶⁾ results indicate oxide stringering with increasing cold work, and the development of a texture. TD-nickel, also severely cold worked after consolidation by extrusion, shows a decided stringering of the oxide particles and a texture. Rasmussen and Grant⁽¹⁵⁾ recorded a well-developed wire texture after severe cold work of low oxide content nickel - ThO_2 alloys. The role played by the texture and the stringered oxides on properties has not been established. Since, almost without exception, test results are obtained parallel to the direction of cold deformation, the properties in the other two directions are poorly known. For example, is the observed increase in ductility after large amounts of cold work plus intermediate temperature annealing^(15,16) of low oxide content alloys

due to the preferred orientation prevailing in the structure or due to a more efficient distribution and rearrangement of dislocations. Clearly the role of texture and its effect on properties should be studied more broadly.

4) Alloyed Matrices - In very early work with alloyed SAP materials, it was indicated that no benefits were derived due to alloying. The alloy selected for the study was not only alloyed but was a precipitation hardening material. More recent studies reported in the USSR⁽²²⁾ support the claim that alloyed SAP offers no important advantages; again the selected alloys were complex, commercial compositions subject to aging effects.

In the USSR studies, it was observed that at low temperatures the properties were those primarily attributable to the alloyed matrix; at high temperatures the properties were those attributable to the oxide dispersion. Unfortunately, repeated exposures to temperature would lead to overaging of the matrix and precipitation around oxide particles, resulting in decreased mechanical properties due to a coarsening of the initial fine structure.

As an alternative to this approach, the use of solid solution strengthened matrices for oxide dispersion strengthened alloys offers clear advantages. duPont reports an increase of over 50 percent in the stress for a 100-hour rupture life at 1800°F⁽²³⁾. Rasmussen and Grant in preparing thorium dispersion strengthened alloys both of Ni and Ni - 12 percent Mo alloys, made by the same technique, also report an improvement of about 50 percent in the stress for a 100-hour rupture life at 1800°F for the Ni - 12 % Mo alloy over pure nickel⁽¹⁵⁾.

Clearly, the combination of major solid solution alloying plus cold work should yield improved high strength properties. At low temperatures the improved strength values due to solid solution strengthening plus cold work (70,000 psi yield strength for Ni - ThO₂ versus 155,000 psi for Ni-12Mo-ThO₂) are responsible for eliminating one of the most objectionable features of early metal-metal oxide alloys, namely, the low yield and ultimate strength values associated with the unalloyed matrix.

Questions of major importance are concerned with the specific alloying elements used to strengthen the matrix:

a) must one use solute elements with low free energy of oxide formation to maintain stable alloys?

b) if internal oxidation is to be the means of introducing the oxide, how much and which type of solute element can be tolerated before the internal oxidation process fails due to formation of surface or sub-surface oxide skins or coarsening of the oxide particles?

c) if solute elements with high free energy of formation values of the oxide are used, will the inadvertant resultant stable oxide of this element unduly complicate the making of a stable dispersion strengthened alloy due to difficulties in reducing the oxide?

Answers to (a) and (c) are considered in the section below. In the case of (b), Yamazaki and Grant have shown that the addition of nickel to copper (10 to 20 weight percent) containing about 0.7 percent aluminum made internal oxidation of the aluminum extremely difficult⁽²⁴⁾. The nickel decreased the solubility of oxygen in copper sharply and slowed down its diffusion rate by a large margin. As a result, only fair success was achieved in producing strong Cu-Ni-Al₂O₃ alloys for high temperature use. The Al₂O₃ was relatively coarse and coarsened as a function of depth of penetration.

5) Dispersoids Other than Oxides - Because of the greater temperature stability and low solubility of oxides, it is not unusual to find that oxides have received the bulk of attention among the investigators of such alloys.

Ryan and Johnstone⁽²⁵⁾ report a titanium nitride dispersion strengthened chromium made by melting, with considerable improvement over pure chromium in tests at 1800°F (7800 psi versus 20,000 psi for a 100-hour life).

Murphy prepared Ni-TiC alloys by internally carburizing Ni - 6% Ti alloys in powder form and hot extruding the resultant product⁽¹⁹⁾. At 1500°F, the stress for a 100-hour life for the alloy was about 10,000 psi compared to 2000 psi for pure nickel, a strengthening factor of 5, in spite of a fairly coarse and non-uniform TiC dispersion. The TiC particles, considerably finer than 1 micron, were observed to be coarsening slowly after 100 hours at 1500°F. While the 1500°F properties were only fair, the yield and ultimate tensile strength values at room temperature were attractive values of about 120,000 and 150,000 psi, respectively, combined with an elongation value of 16 percent.

As was suggested above, the high values of strength at low temperatures are associated with a contribution of bonding energy between the two phases of the alloy in contrast to the lack of bond strength between oxides and the metallic matrix. Developments involving aspects of both sections 4 and 5, above, appear attractive. Properly alloyed matrices of a solid solution nature, combined with carbides, borides, aluminides, and beryllide dispersoids may be expected to yield useful combinations of low and high temperature strength values with good temperature and time stability. Considerations of particle size and volume content of the hard particle would follow experience largely gained from oxide dispersion strengthened alloys.

II. Structure Stability

1) Oxide Coarsening - That oxides, known to be insoluble in specific metallic matrices, coarsen at high temperatures came as a shock to those who expected unlimited structural stability in metal-metal oxide alloys. Figure 2 shows the decrease in room temperature hardness of a Cu - SiO₂ alloy (prepared by internal oxidation) as a function of the one-hour annealing temperature⁽²⁾. A drop in hardness is observed above about 700°C (1290°F). In Figure 3 are electron-micrographs showing the resultant increase in size of the SiO₂ (cristobalite)⁽²⁶⁾.

It has been suggested by some that solution of SiO₂ takes place; others have implied that there is slow but definite reduction of SiO₂ by the copper at these high temperatures. If these speculations were correct, it would be much harder to explain the coarsening of Al₂O₃ in copper, or ThO₂ in nickel. Further, one would have to explain why copper can be melted in SiO₂ or Al₂O₃ crucibles, and held for hours many hundreds of degrees hotter, in the liquid state, than the observed coarsening temperature of 700°C (1290°F), without pick-up of significant aluminum or silicon by the copper; thermodynamically one cannot expect reduction of these oxides by copper.

If SiO₂ was soluble in copper, one would expect that coarsening of the oxide would be controlled by diffusion of the silicon or oxygen; the heat of activation for the process would be expected to be between about 40,000 and 50,000 calories per gram atom. Komatsu and Grant made measurements of the coarsening of SiO₂ and calculated a heat of activation value for the coarsening process⁽²⁶⁾. When the SiO₂ particle size was 0.5 micron, ΔH (activation) was about 80,000 calories; when the particle size was 1.0 micron, ΔH was more than 100,000 calories. Clearly the coarsening process is much more difficult than would be expected if diffusion of Si and O₂ was

the controlling process. Further, the coarsening process becomes more difficult as the SiO_2 particle size increases. These observations suggested that the controlling step in oxide coarsening is the decomposition pressure of the SiO_2 at any temperature. As the particles coarsen, the total surface area decreases as the square of the diameters, sharply decreasing the potential for escape of dissociated silicon and oxygen ions to enter into solution in the copper⁽²⁶⁾.

In contrast, if one considers a system such as $\text{Ti-Al}_2\text{O}_3$, it was shown in the author's laboratory that in a powder mix of this sort (titanium was introduced as 5 microns titanium hydride, and Al_2O_3 as 10 volume percent of 0.03 micron gamma alumina), even at an extrusion temperature as low as 982°C (1800°F) about 80 percent of the Al_2O_3 was reduced. In this case one accepts reduction because of the nearly similar free energies of formation of the respective oxides.

2) Phase Transformations - If a phase transformation takes place either in the oxide or matrix phase, the tendency is to change the level of stored energy in the system. In the event that the transformation takes place in the metal, recrystallization occurs and all the stored energy of deformation is lost. This was the case with $\text{Fe-Al}_2\text{O}_3$ alloys extruded in the high temperature gamma-iron field⁽²⁷⁾.

If the transformation takes place in the oxide phase, a volume change takes place which may result in an increase or a decrease in size, but with a change in the strain energy around the particle. Gamma Al_2O_3 changes to alpha, the stable high temperature form at temperatures as low as 700°C (1290°F) under conditions of pressure and the presence of non-refractory oxide impurities⁽²⁸⁾. There is a volume decrease and a loss of properties. Further, Predecki and Grant observed that Ni-ZrO_2 alloys

suffered a sharp decrease in hardness at about 1000°C (1820°F) due to a transformation of ZrO_2 at that temperature⁽¹²⁾.

The presence of other oxides, particularly non-refractory oxides, as contaminants (Fe_xO_y , NiO , Cu_xO_y) has been observed to increase the coarsening rate of refractory oxides by decreasing the decomposition temperature of the latter⁽²⁸⁾.

III. Current Trends

In line with observations made above, research and development efforts today are concerned with the most refractory oxides, free of transformations (ThO_2 , BeO , MgO , CaO), plus alloyed matrices. Because oxide dispersion strengthened alloys show their best properties at the higher temperatures and longer future times (see Figure 4⁽²⁹⁾), it is altogether too obvious that oxidation resistant alloys are required. While coatings are a possibility for new stronger alloys which may be developed, coatings have not indicated any large measure of success in the past.

Chromium (above about 20 percent) and aluminum (above about 7 percent) are the best elements for endowing oxidation resistance. Processing methods must be capable of introducing these elements in the desired amounts without accompanying oxidation because reduction of Cr_2O_3 is extremely difficult and of Al_2O_3 is impossible; without damaging the alloy structure. Whether chemical means or the more direct powder approaches will yield better results is pure speculation at this time.

More complex alloy matrices will be examined. The presence of significant (though small) amounts of reactive elements, such as Al, Ti, Zr, Be, Mg, Y, Ta, Cb, Si, etc.,) will introduce problems not unlike that already encountered in the case of chromium additions, but will also raise the question of subsequent interaction of these reactive elements with

the refractory oxide.

To obtain other classes of alloys capable of very high strength at low temperatures, high strength properties at intermediate temperatures (up to 1600°F), and fair strength and stability above 1600°F, may demand closer and more extensive study of dispersoids other than the oxides.

It is clear that oxides, in spite of very high melting points and apparent temperature - time stability in bulk form (ceramics), have an upper temperature - time limit well below the melting temperature of the oxide. The matrix is an important factor in determining the oxide stability. It certainly does not appear now that long-time stability can be expected much above about 1500 or 1600°C (2732 to 2912°F), if the major concern is high strength. A large measure of structural stability can be expected above these temperatures, but strength benefits will largely be lost if the particle spacing exceed about 1 micron.

Nevertheless, because oxidation resistant alloys are not now available (other than platinum - ThO₂) above about 1800°F (982°C), and conventional, aged high strength alloys are not available in Fe, Co, Ni, Cr alloys above about 2000°F (1093°C), there is much room for development and growth.

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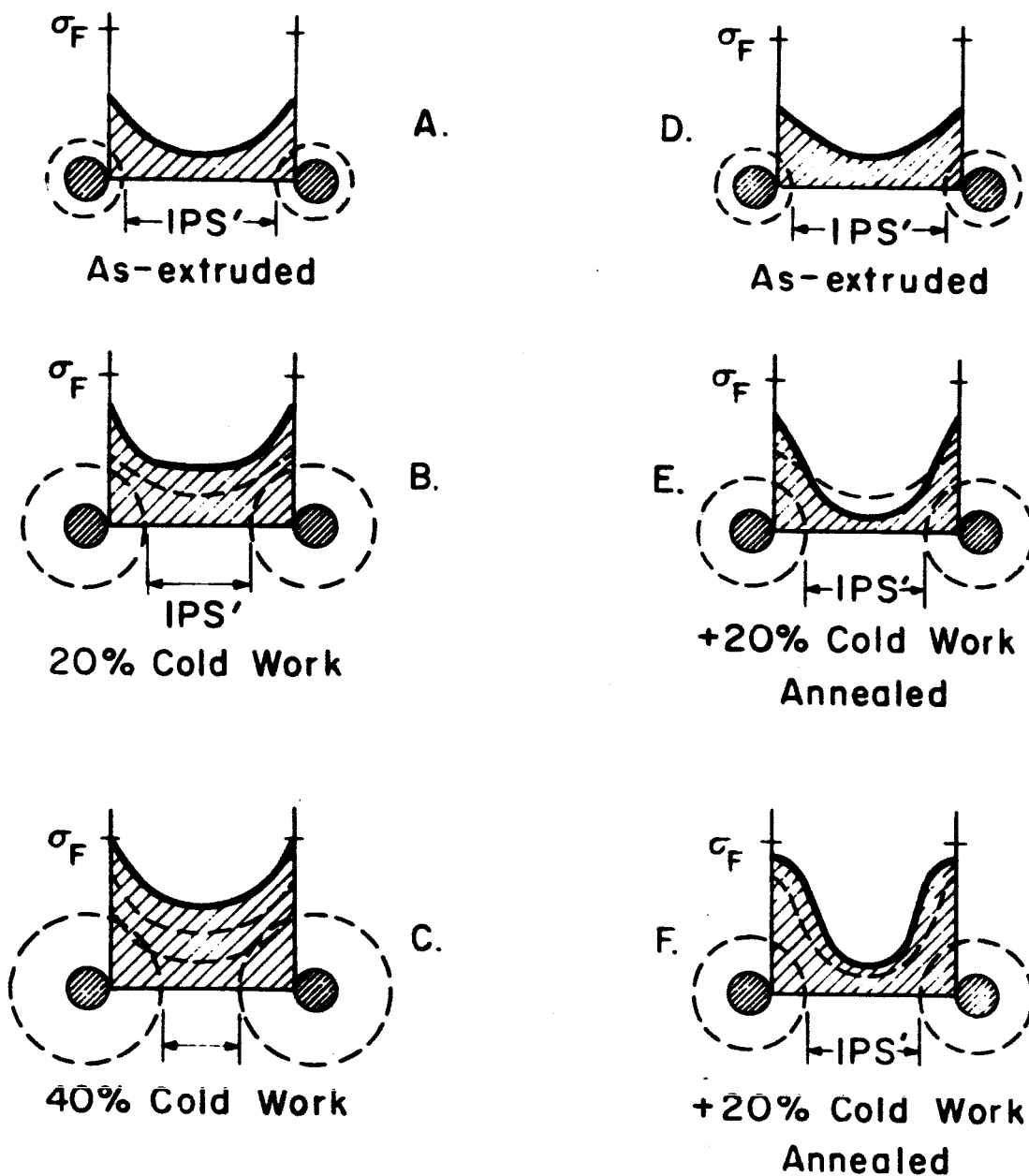


Figure 1. Interparticle and "apparent" interparticle spacing⁽¹⁵⁾ among oxide particles for (σ_F is fracture stress):

- (a) left series: increasing cold work
- (b) right series: increasing cold work plus intermediate annealing treatments. At stage F, there is an additional increment of 20% cold work over stage E.

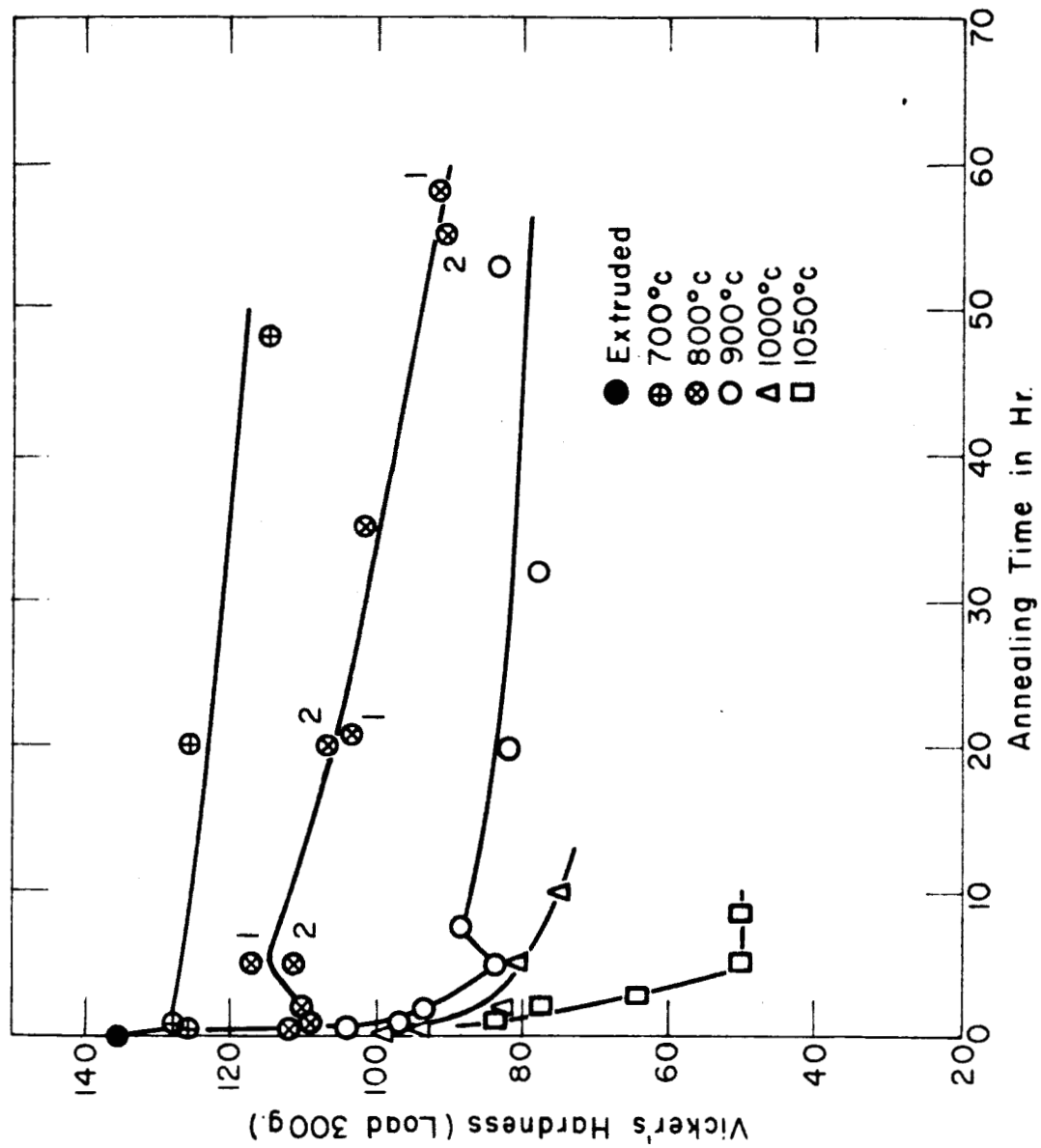


Figure 2. Decrease in hardness with annealing time (26)
from 700° to 1050°C.

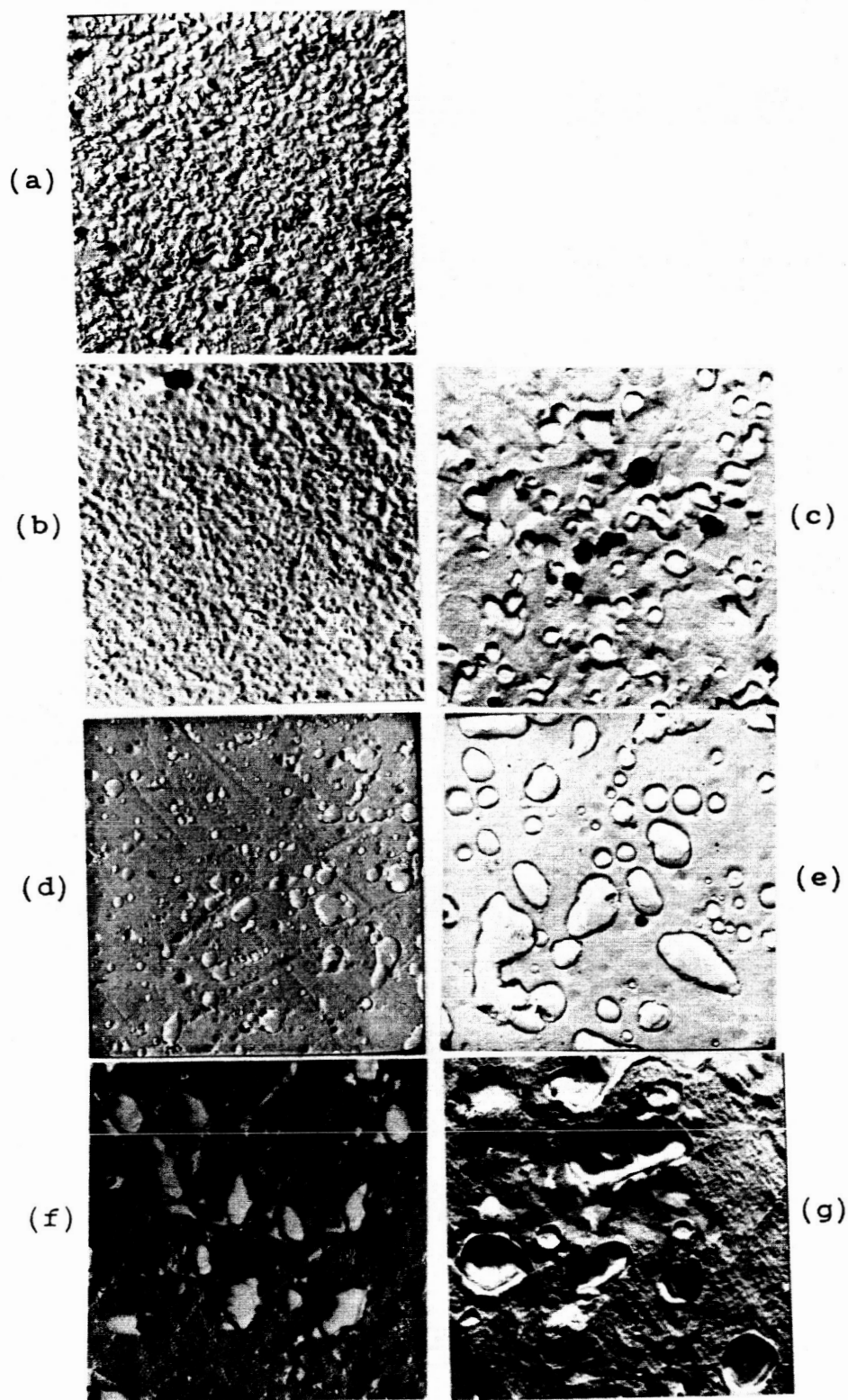


Figure 3 Surface replica electron micrographs of the Cu - 12 v/o SiO₂ alloy after various heat treatments. Unetched. 5000X.

(a) As extruded	(b) 800°C, 5 hrs.	(c) 800°C, 20 hrs.
(d) 900°C, 2 hrs.	(e) 900°C, 20 hrs.	(f) 1000°C, 2 hrs.
(g) 1000°C, 5 hrs.		

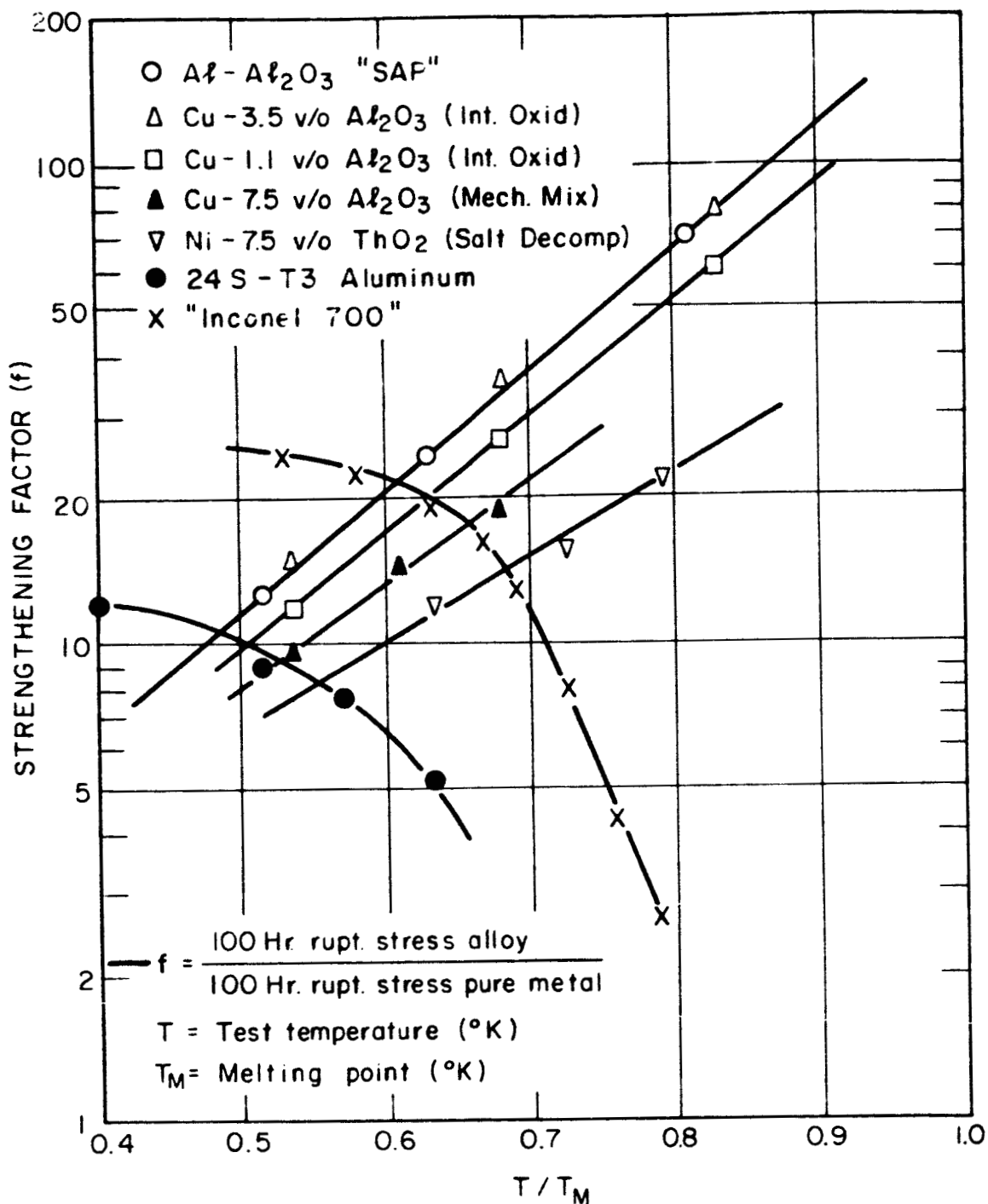


Figure 4. Strengthening factor versus homologous temperature for a series of oxide dispersion strengthened alloys compared to two conventional wrought alloys.